

MULTI-STAGE AP MECHANICAL PULPING WITH REFINER BLOW LINE TREATMENT

5 REFERENCE TO RELATED APPLICATIONS

The present application is a continuation-in-part of International Application No. PCT/US0223078 under 35 U.S.C. §365(c) filed July 19, 2002 (designating the U.S.) which claims benefit under 35 U.S.C. §119(e) of United States Provisional Application No. 10 60/306,974 filed July 19, 2001.

FIELD OF THE INVENTION

The present invention relates to a process for the production of pulp from lignocellulosic material, such as wood chips or the like, by 15 chemical-mechanical refining.

BACKGROUND OF THE INVENTION

Applying alkaline peroxide chemicals in a mechanical pulping system (APMP) may be traced back as early as 1962. Since then, 20 there have been a number of different process ideas developed to apply the chemicals before or during early stages of refiner pulping. In recent years, an extensive and systematic investigation has been reported on how different chemical treatments in refiner mechanical pulping affect pulp property development and the process 25 consumption. For hardwoods, it was observed that alkaline peroxide pretreatment in general gives better optical properties, better bleachability and higher pulp yield at similar strength properties when compared to other conventional chemical pretreatment, such as alkaline sulfite and cold caustic soda processes. When compared to a peroxide post-bleaching process, applying alkaline peroxide before 30 refining has a tendency to give a higher bulk at a given tensile strength for some hardwood species, such as North American aspen.

In a very broad sense, alkaline peroxide refiner mechanical pulping is a type of pulping process where hydrogen peroxide and alkali in various forms, together with various amounts of different peroxide stabilizers, are applied to the lignocellulosic materials before or during defiberization and fibrillation in a refiner. In the early stage of development of this type of pulping process, two basic concepts were tried. One was to apply alkaline peroxide treatment on chips, to allow the bleaching reactions to complete or to approach completion before refining. The other basic concept was to apply all the alkaline peroxide at the refiner, either with no pretreatment or with stabilizers or other alkaline pretreatment prior to the alkaline peroxide application at the refiner.

Conventionally the inclusion of chemicals such as silicates prior to the refiner leads to a situation where scale forms on the processing equipment. The refiner area itself also can suffer due to the formation of silicate precipitates, especially in processing softwoods, which can lead to a glassing of the refiner plates.

The application of chemicals at a point downstream of the refiner has also been proposed. However these proposals did not encompass the use of chemical pretreatment or conditioning of the chips. In addition such downstream chemical addition appeared incompatible with high pressure refining conditions.

Summary Of The Invention

The present invention is directed to the introduction of chemicals to lignocellulosic material immediately after refining in order to achieve, among other things, a comparable bleaching efficiency as when applying chemicals at locations upstream of and/or at the refiner.

The introduction of chemicals downstream of the refiner, wherein the refiner may be a primary, secondary and/or tertiary refiner, is utilized with the concept of applying chemicals such as alkaline peroxide pre-treatment to lignocellulosic material before

refining. Preferably, the refiner has a highly pressurized case, for achieving the known benefits of high pressure refining.

The introduction of chemicals downstream of the refiner according to the invention may alternatively be utilized with the process referred to herein as P-RC (Preconditioning followed by Refiner Chemical treatment) for APMP, which combines the concept of applying chemicals such as alkaline peroxide as a pretreatment to lignocellulosic feed material before primary refining with the concept of applying chemicals such as alkaline peroxide at the primary refiner.

The preferred embodiment of the invention includes applying more than one-third of total alkaline peroxide (and/or other chemicals known in the art to bleach or otherwise process lignocellulosic material into pulp or precursors of pulp) at or near the blow valve in the post refiner intermediate line, in combination with chemical addition at the refiner and chemical impregnation of the chips upstream of the refiner, to yield a more energy efficient process and to allow a more efficient bleaching than the application of all the chemicals before discharge from the refiner.

A significant benefit of the invention is better chemical efficiency, by moving a greater number of chemical reactions downstream relative to conventional techniques, resulting from the relatively heavier or more intense addition of chemicals and/or chemical stabilizers at the post refiner blow line.

A further benefit of the invention is the reduction in the detrimental effects of the high temperature and/or other conditions prior to and during high pressure primary refining, which are known to influence pulp brightness and development.

Another benefit of the invention as implemented in a high-pressure system, is the recovery of more and higher quality of steam and/or heat than in other types of P-RC APMP systems, where the primary refiner is either completely atmospheric or atmospheric at the inlet.

Brief Description Of The Drawings

The invention will be better understood by reference to the accompanying drawings in which:

5 Figure 1 is a block diagram depicting the general P-RC APMP process.

Figure 1A is a block diagram depicting steps of transferring lignocellulosic material to a refiner having a casing at atmospheric pressure, with discharge at atmospheric pressure.

10 Figure 1B is a block diagram depicting steps of transferring lignocellulosic material to a refiner having a pressurized casing with pressurized discharge.

15 Figure 1C is a block diagram depicting steps of transferring primary pulp produced in the refiner with a casing at atmospheric pressure, to a high consistency tower via a transfer device.

Figure 1D is a block diagram depicting steps of transferring primary pulp produced in the refiner with a casing at atmospheric pressure directly to a high consistency tower.

20 Figure 1E is a block diagram depicting steps of transferring primary pulp produced in the refiner with a pressurized casing, to a high consistency tower via a transport device.

Figure 1F is a block diagram consistent with an embodiment of the invention, depicting steps of transferring primary pulp produced in the refiner with a pressurized casing to a high consistency tower.

25 Figure 2 is a table comparing P-RC with two prior art processes.

Figure 3 is a graph of freeness as related to energy consumption for P-RC and two prior art processes.

30 Figure 4 is a graph of density as related to energy consumption for P-RC and two prior art processes.

Figure 5 is a graph of the tensile of tensile development for P-RC and two prior art processes.

Figure 6 is a graph of burst development for P-RC and two prior art processes.

Figure 7 is a graph of brightness development for P-RC and two prior art processes.

5 Figure 8 is a graph of the light scattering coefficient of the pulp as a function of freeness for P-RC and two prior art processes.

Figure 9 is a comparative table of atmospheric versus pressurized casing processing of aspen wood chips according to P-RC.

10 Figure 10 is a comparative table of atmospheric versus pressurized casing processing of birch wood chips according to P-RC.

Figure 11 is a block diagram consistent with an embodiment of the invention, depicting steps of transferring primary pulp produced in a refiner with a pressurized casing to a retention tower with a chemical addition in the intermediate line following the control valve.

15 Figure 12 is a block diagram consistent with an embodiment of the invention, depicting steps of transferring primary pulp produced in the refiner with a pressurized casing to a retention tower with an alkaline peroxide chemical addition in the intermediate line prior to the inlet of the separator.

20 Figure 13 is a block diagram consistent with an embodiment of the invention, depicting steps of transferring primary pulp produced in the refiner with a pressurized casing to a retention tower with an alkaline peroxide chemical addition in the intermediate line at the separator.

25 Figure 14 is a block diagram consistent with an embodiment of the invention, depicting steps of transferring primary pulp produced in the refiner with a pressurized casing to a retention tower with an alkaline peroxide chemical addition in the intermediate line at the separator discharge.

Figure 15 is a comparative table of refiner eye versus blow line chemical addition processing of birch and maple wood chips according to the invention.

5 Figure 16 is a comparative table of refiner eye versus blow line chemical addition processing of spruce and red pine wood chips according to the invention.

Figure 17 is a comparative table of refiner eye versus blow line chemical addition processing wood chips at higher pressure according to the invention.

10 Figure 18 is a block diagram consistent with an embodiment of the invention, depicting steps of transferring pulp produced in a pressurized refiner via a intermediate line to a tower.

Detailed Description Of The Invention

15 Figure 1 presents a simplified process flow diagram of the P-RC alkaline peroxide mechanical pulping (APMP) process. The P-RC process generally applies alkaline peroxide chemicals at chip pretreatment/chip impregnation step(s)/stage(s) 1, 2 and as the material is fed to the primary refiner 3.

20 The preconditioning step(s) as implemented in stages 1 and 2 of Figure 1, preferably include one or two atmospheric compression devices, such as screw presses. Chip material is fed through an inlet, and passes through at least one compression region and at least one expansion region, and is discharged. A chemically active solution (pretreatment solution) is added to the material, typically while decompressing or decompressed at or near the discharge to facilitate penetration of the solution into the material.

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30 The refining step 3 may include a primary refiner of conventional size, configuration, and operating conditions as known for chemi-mechanical pulping. Depending on such factors as whether chemicals are to be added and what types of chemicals if any are to be added the size, configuration, and operating of the refiner can be

tailored so as to not expose the chemicals to excessive temperature or time-temperature combination. In one embodiment of the invention the pressure can be within a range of about 15 psi to pressures greater than 45 psi. Any chemicals added at the refiner
5 will be referred to as the refiner solution.

Steps implemented following the primary refining, may have a level of chemical presence carried downstream from the refiner or other upstream processing. In one embodiment of the invention, the post refining chemical environment is modified by an addition or
10 additions of a intermediate line solution or solutions to the intermediate line. The intermediate line is located between the refiner and the retention tower. For instance, as shown in Figure 11, alkaline peroxide solution is applied to pulp in the intermediate line, at the blow line 30, after exposure to and discharge from the refiner. The chemicals
15 may be applied at a point or points along and about the blow line 30. The blow line 30 may extend between the blow valve and a separator of the intermediate line. As shown in Figure 18, the chemicals may also be applied in the intermediate line immediately after the blow valve 40, between the blow valve and the separator 42 immediately prior to separator 44, at the separator 46 and/or immediately after the separator
20 48. The separator, for instance a cyclone, may operate to separate steam/heat/liquid or combinations of those items from the pulp. Prior to entry into the separator the pulp may have a consistency of about 20% to about 60% and a temperature of about 80°C to about 155°C.
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Injection of the chemicals at a intermediate line location or locations may be made through simple orifices in the intermediate line and/or by the use of injectors, such as nozzles, associated with the line. The nozzles can be associated with the intermediate line in various ways along and about the intermediate line to desirably control the chemical addition. The control can be dependent, for example, on the effect that the additions have with regard to the bleaching process and/or conditioning process. Chemical profiles within the pulp flow can thus be modified or maintained by, for example, injection sequencing, flow rate,
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composition, and/or duration. Other variables such as the depth of injector intrusion into the flow path, injector angle, injector orifice configuration, and other properties of the injector installation may be modified to achieve a desired result. Chemical introduction may be
5 modified by varying the introduction location based on the pressure used in refining. For instance, alkaline peroxide chemicals may be introduced immediately (from less than a few inches to a few feet) after the blow valve, especially in low pressure refining where the pressure is less than about 45psi. The alkaline peroxide chemicals may also be introduced immediately before the cyclone (from less than a few inches to a few feet) after the blow valve, especially in high pressure refining where pressures higher than 45psi are used. In other cases the alkaline peroxide chemicals may be introduced intermediate the cyclone and the blow valve, or even at the cyclone.
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15 The refiner may be primary, secondary, and/or tertiary, with a pressurized casing or fully pressurized from preheater to refiner discharge. The pressure in the refiner aids in expelling the pulp from the refiner during discharge. The discharge can be modified or controlled by, e.g., the blow valve. The pressure assisted discharge of the pulp into the
20 intermediate line can result in the pulp having a residence time of a few seconds to minutes in portions of the intermediate line. The pulp can achieve high velocities and experience significant turbulence as it flows through the intermediate line. These conditions enhance the mixing between the chemicals and the pulp. The intensive turbulence and a high temperature gradient in the pulp stream may also assist in transferring the chemicals to individual pulp fibers as well into the fiber wall.
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30 As an illustrative example, the pulp may be about 100 °C or higher, and the chemical liquor may be 40°C or lower. The intermediate line solution may preferably be in the range of about 10°C to about 25°C but can be up to 80°C. The application of alkaline peroxide chemicals at the intermediate line reduces the exposure time of the alkaline peroxide chemicals to high temperature, especially when elevated temperature and/or pressure is present at refining. This post refining addition to the pulp flow through injection proximity, facilitates an easier stabilization and

an increased efficacy of the peroxide. The use of the invention in an intermediate line with a superatmopheric refiner system also can result in the enhanced or modified recovery of steam/heat/liquid from the pulp. Such steam may be diverted away through a steam pipe 36. These
5 features also allow for the production of high-freeness pulps with low shives content, since it is well known in the industry that the higher refining pressure tends to produce lower shives, or cleaner pulp. In some cases a press may be included in addition to or in place of the cyclone 32. The press could allow for an increase in steam/heat/liquid recovery
10 from the pulp.

In one embodiment of the invention the optimizing process to influence peroxide efficiency and brightness development can be accomplished when the primary refining is fully pressurized. In one particular configuration this may be referred to as P-RC APTMP,
15 which differs from other P-RC APMP configurations where the primary refiner is operated either under completely atmospheric pressure, or with atmospheric pressure at the inlet and low pressure at the casing.

Figures 1A through 1F present various examples of a P-RC process of the type generally shown in Figure 1. For example,
20 Figures 1 A and B show that after the material is pretreated at 1 and/or 2, addition of the solution to the lignocellulosic material may more specifically occur at a cross conveyer 10, downstream of the screw press and near refiner 3, or at the refiner itself, e.g., the ribbon feeder 12, the inlet eye of the refiner disc 14, and/or at the inlet zone
25 of the plates on the refiner disc 16. As used herein, chemical addition "as the material is fed to the refiner", encompasses the locations 10, 12, 14, and 16. The refiner in a P-RC process may have an atmospheric casing 3A or an overpressure casing 3B, but the inlet to the refiner would normally be at atmospheric pressure. The discharge from a pressurized casing 20a of primary pulp may be
30 through a blow valve or similar device, and discharge from an

atmospheric casing 20 may be by gravity drop or the like. The discharge from the refiner will, in any event, directly or indirectly go to a high consistency-bleaching tower 24 of any type known in the art (but subject to temperature control).

5 In one embodiment of the invention the pretreatment solutions, the refiner solutions (if present), and the intermediate line solutions act chemically on the lignocellulosic material. It may be advantageous, depending on the lignocellulosic material and the processing equipment, to modify the chemical exposure profile of the
10 material to the chemical agents in order to optimize the process, and/or eliminate or reduce unwanted chemical effects or degradation. Such chemical profile modification may be accomplished by sequential chemical additions throughout the process, and can be combined with other variable conditions such as temperature,
15 concentration, pressure, and duration to further enhance the desired effect.

20 Lignocellulosic material processed using the P-RC process can be discharged 4 from the primary refiner casing (either atmospheric discharge 20 or overpressure discharge 20a), as a primary pulp having a measurable freeness and could properly be called a pulp able to form a handsheet. As shown in Figures 1 C and D, atmospheric discharge from the refiner could pass via a transfer device 22 such as a transfer screw, to the tower 24, or more directly 28 via a chute or the like. As shown in Figures 1E and F, with a pressurized casing the
25 refined pulp would typically be discharged through a blow valve and delivered either directly or indirectly to the tower. Optionally, as shown in Figures 1C and E, the bleached pulp exiting the tower can be further processed in, e.g., a secondary refiner. The high consistency retention tower 24 allows the chemical bleaching reactions carried over from upstream of the tower to continue.
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In one embodiment of the invention, for example as shown in Figure 18, the discharge from the blow valve may be delivered indirectly to a retention tower through a separator and/or a press.

The presence of an ample amount of the alkaline peroxide chemicals in the primary refiner (e.g., as by shifting a large proportion of the chemical reactions to the refiner chemical treatment stage) improves efficiency. This is because variations in chip forms and quality, in addition to the natural heterogeneity of wood chips and fibers, often make it difficult, if not impossible, to achieve a good chemical distribution in the chip pretreatment/impregnation stage(s). In these situations, the mixing action at the primary refiner helps to promote chemical distribution, and hence, improves the chemical efficiency.

In accord with one embodiment of the invention, the addition of chemicals into the post refining intermediate line allows, for example, the use of a pressurized refiner and higher temperatures in refining. Addition of chemicals to the intermediate line at, for example, the blow line provides for a fast, and more direct, distribution of chemicals such as peroxide to the chromophore sites for efficient bleaching. This efficiency is achieved because the targeted peroxide reactions are carried out at the reaction site of interest quickly without lengthy exposure to the more heterogeneous environment present in previous portions of the process. Conventionally the temperature at the inlet between the plates of a refiner pushes the chromophore removal and hemicellulose alkali reactions so fast that that pH is lowered prematurely. Using the post refiner intermediate line as the location for chemical mixing according to an aspect of the present invention, distributes the chemicals fast enough, to compete favorably against and counter to a significant extent, the elevated temperature of the pulp. Such elevated temperature can be, for example, from about 80°C to about 155°C.

In one embodiment of the invention, the pulp can be maintained in an interstage high consistency retention tower. The pulp in the high consistency retention tower may have a consistency of about 20% to a consistency of about 40% consistency, with a preferable consistency of about 30%. The temperature of the pulp in the high consistency retention tower may be from about 60°C to about 95°C. The pulp can be held in the retention tower from about 5 30 minutes to more than 2 hours depending on the chemical reaction needed for chemical treatment. The maintenance conditions include 10 but are not limited to temperature, pressure, pH, chemical concentration, solids concentration, and time, that allow for conditioning and/or bleaching of the pulp to continue and limit the degradation of the bleaching agent through reactions that are extraneous to the bleaching of the pulp. Such extraneous reactions 15 may be non-productive, inefficient, and/or harmful to the bleaching of the pulp. Control of some and/or all of the conditions may or may not be needed depending on e.g., the type and condition of the lignocellulosic material used in the process, and the type, size and operating environment of the equipment itself. For example, 20 conditions of temperature may be modified throughout the process by the addition of the chemicals, pressurized gas, and other heating or cooling methods. Temperature modifying means may be employed during transfer of the primary pulp 22 by using a mixing screw with water added while the pulp is mixed and transferred to the tower. 25 The temperature of the primary pulp may also be thermally adjusted within the tower if the primary pulp is discharged directly to the tower 28, by means known in the art. For example, the pulp may be thermally adjusted through addition of liquids or gases, and/or through use of heat transfer components such as tubing, tower jacketing, etc.

As used herein, the term "control" should be understood as including both active and passive techniques. Thus, control could be

implemented by a static hardware configuration or by continually measuring one or more process parameters and controlling one or more process variables.

The chemical conditions present anywhere in the inventive process may be modified by additives to prevent extraneous degradation. This modification may be made at, by way of example, the pretreatment step(s) 1 and/or 2, the cross conveyer 10, the ribbon feeder 12, the inlet eye of the refiner disc 14, the plates of the refiner disc 16, the blow valve 20a, the blow line 30, the separator, 10 32, and/or after the separator. An example of stabilizers would be chelation agents. A chelation agent refers to a compound that has an ability to form complexes, so called chelates, with metals occurring in the lignocellulosic material, and primary pulp. Such metals may include monovalent metals sodium and potassium, earth-alkali divalent metals calcium, magnesium and barium, and heavy metals such as iron, copper and manganese. The metal ions retained in the material as it is processed makes the bleaching by oxygen chemicals (such as hydrogen peroxide) less effective, and results in excess chemical consumption as well as other problems well known 15 in the art. In order to reduce or eliminate the effect of these metal ions on the process, chelants such as for example diethylene triamine pentaacetic acid (DTPA), ethylene diamine tetraacetic acid (EDTA) and nitriletriacetic acid (NTA) may be used. These and other chelation agents known in the art may be used alone or in combination as needed or desired depending on process conditions. In addition, silcates and sulfates as examples may also be used advantageously as stabilizers as well as serving other functions well 20 known in the art.

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Further embodiments and aspects of the invention will be apparent from the examples and description set forth below.

ILLUSTRATIVE EXAMPLES

Example Set A

5 Several general series of pilot plant processes are illustrated in the following examples. The materials and conditions for the following examples, unless specified otherwise are:

10 **Wood:** A blend of 50% aspen and 50% basswood was used in this study. The aspen woods had rotten centers, which made it more difficult to bleach than normally expected. The woods were all from Wisconsin USA, and debarked, chipped and screened before further processing.

15 **Chemical Impregnation:** Chips were pre-steamed first for 10 minutes, and then pressed using an Andritz 560GS Impressafiner at 4:1 compression ratio before impregnated with alkaline peroxide chemical liquor. The chemical liquor was introduced at the discharge of the press, and allowed for 30 minutes retention time before refining.

20 **Refining:** An Andritz 92 cm (36") Model 401 double disc atmospheric refiner at a conventional speed of 1200 rpm was used for all the refining processes. There was 15 minutes or more retention time between the primary and the secondary, and no dilution after the primary and before the secondary. The refining consistency was 20% at both the primary and the secondary.

25 **Pulp Testing:** Tappi Standards were used for all pulp testing except for freeness, which follows Canadian Standard Freeness (CSF) test methods.

30 In the first of three processes compared, all of the alkaline chemicals were applied, (3.3% total alkalinity, (TA), and 2.4% H₂O₂,

together with 0.2% DTPA, 0.07% MgSO₄ and 3% Na₂SiO₃) at the chip impregnation (preconditioning or pretreatment) stage, (only one stage chip impregnation was applied), then refined at atmospheric pressure. This series was, therefore, named "Chip". The second series used approximately two thirds of the total alkaline peroxide chemicals, (or 2.4% TA, 1.6% H₂O₂, 0.08% DTPA, 0.04% MgSO₄ and 2.4% Na₂SiO₃), at the chip impregnation stage, and approximately one third of the total chemicals, (1.0% TA, 1.0% H₂O₂, 0.19% DTPA, 0.05% MgSO₄, and 0.9% Na₂SiO₃), at the eye of the primary refiner. It is labeled as "Chip + Refiner", and represents the invention. In the third series, labeled "Refiner", the chips were first pressed using the same chip press as the first two series, and then all the alkaline peroxide chemicals, (4.2% TA, 3.3% H₂O₂, 0.36% DTPA, 0.11% MgSO₄, 4.3% Na₂SiO₃), were applied at the eye of the primary refiner. In all the series, the pulp from the primary was allowed 15 minutes retention under cover in drums, (which gave a temperature about 80-90°C), before the second stage refining. There was no interstage washing.

Figure 2 summarizes some of the process conditions and results from each series. The pulps are all from second stage refining. In peroxide bleaching of mechanical pulps, a lower TA/H₂O₂ ratio is in general preferred under higher temperature to prevent, or to reduce the possibility of alkali darkening reaction. For this reason, as shown in Table 1, the lowest TA/H₂O₂ ratio, 1.27, was use for "Refiner" series, the second lowest, 1.31, for "Chip + Refiner" series, and the highest, 1.37, for "Chip" series. In "Refiner" series, a larger amount of TA charge (4.2%) was used to prevent pH from dropping too fast and too low during refining because of the high temperature and the heat generated from refining energy. Reasonable amounts of residual peroxide and pH were maintained in each of the series, Figure 2.

As to the chemistry, the main difference between "Chip" and "Chip + Refiner" series is that the latter is more aggressive in moving more alkaline peroxide chemicals to the refiner chemical treatment stage.

Graphic presentation of the data gathered from pulp after secondary refining after different investigated processes are shown in Figures 3 through 8. Figure 3 shows effects of the different chemical applications on pulp freeness development in relation to specific energy consumption (SEC), which includes energy consumed during chip pretreatment stage. The "Chip + Refiner" series used slightly less SEC than the "Chip" series, but both series used, on average, approximately 200 kwh/odmt less SEC than the refiner bleaching series, "Refiner", even though the latter had more caustic chemicals applied than the first two series and has the same residual pH, 8.2, as "Chip + Refiner" series. It appears that adding the alkaline chemical under high temperature, at refiner eye, causes more alkali consumed on nonproductive, or side reactions that have little to do with pulp property development.

It should be pointed out that in a commercial operation, the SEC in general is lower than that observed at the lab for chemical mechanical pulping of hardwoods. The SEC values in Figure 3, therefore, are better used for comparison purpose than for their absolute values.

Because many pulp properties, especially the strength properties, are dependent on handsheet density, this property was also analyzed under SEC, and results are shown in Figure 4. In this case, the more aggressive refiner chemical treatment P-RC APMP series, "Chip + Refiner", had the best efficiency for handsheet density development, which was followed by "Chip" and "Refiner" series. These results demonstrate that in chemical mechanical pulping, process energy efficiency depends not only on how much but also on how the chemicals are applied.

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As for pulp intrinsic property development, there was however, little difference among the three series, as illustrated in Figures 5 and 6, suggesting that as long as the chemicals are added before refining, the mechanism involved in fiber strength property development remains the same.

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As for pulp optical property development, in mechanical pulping, pulp brightness is often freeness-dependent. Figure 7 shows brightness at different freeness from each series. Of interest is that "Chip + Refiner" series had a similar brightness development as that of the "Refiner" series, even though the former used less amount of the bleaching chemicals, 2.6% H₂O₂/3.4% TA versus 3.3% H₂O₂/4.2% TA. Adding all of the chemicals at the impregnation stage, "Chip" series, showed also a less bleaching efficiency, 2 or more points lower, than that of "Chip + Refiner" series. This suggests that the bleaching efficiency is sensitive to how the chemicals are distributed between the chip impregnation and refining in P-RC APMP process. In this case, a compromise between adding all of the chemicals at chip impregnation or at eye of refiner appears to be the most efficient in bleaching and peroxide consumption.

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Figure 8 shows that there was no difference in light scattering property development in all the series studied, suggest the pulp surface development mechanism also remain the same as long as the chemicals are added before refining.

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Example Set B

The below examples illustrate a different refining configuration where the primary refiner was maintained at a negligible gauge pressure at the inlet and a low pressure (approximately 140 kPa) at the casing. Advantages of this configuration include:

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- 1) better steam handling at the refiner discharge, especially for high capacity refiners (300 t/d or higher);

2) ease of transfer primary pulp from the refiner to the interstage high consistency (HC) tower;

3) a potential to use some of the steam generated from the primary refining (by using a cyclone to separate steam and pulp fiber);

5 4) ease of converting existing TMP systems into a P-RC APMP process.

These examples show that running the primary refiner at a low pressure (140 kPa) in the casing and atmospheric at the inlet can give similar bleaching efficiency as that of atmospheric at both the inlet and the casing. Temperatures at the inlet and between the plates in the primary refiner may push the chromophore removal and hemicellulose alkali hydrolysis reactions fast enough that pH was lowered considerably before the pulp reaches the casing out off the refiner plates. The pulps at the cyclone discharge from the primary refiner were measured in the examples below to have pH of 9.3-9.7, at which peroxide is easy to stabilize even under the high temperatures (80-90 °C) observed.

The materials and conditions for the following examples below were as follows:

Wood: Aspen and birch chips from a commercial pulp mill in eastern Canada were used in this study.

Chip Impregnation: A conventional pilot chip impregnation system was used in this study. In all the P-RC APMP runs studied, only DTPA was used in the first stage of chip impregnation. The chips were then impregnated with alkaline peroxide (AP) chemicals at second stage impregnation. The AP treated chips were then allowed for 30 to 45 minutes' retention (without steaming) before being refined.

Atmospheric Refiner System: Andritz 36" diameter (92 cm) double disc 401 system is typically used for conventional P-RC APMP

process investigations. This system consists of an open metering belt, an incline twin-screw feeder, the refiner and an open belt discharge. The system is used for both primary and later stages of refining. When used for the primary, the pulp discharged were collected in drums and kept under cover to maintain a high temperature (typically 80 to 90 °C) for a certain period of time.

Pressurized Refiner System: An Andritz single disc 36" diameter (92 cm) pressurized system was modified for atmospheric inlet/pressurized casing configuration. The original refiner system has all the standard features of a conventional TMP system. In order to run the system with atmospheric pressure at the inlet, a valve was placed on top of the vertical steaming tube and was kept open during refining. During the trial, the plug screw feeder (PSF) was run at 50 rpm (normal speed for TMP is 10 to 20 rpm) to ensure the chemical impregnated chips were not compressed. The AP impregnated chips were placed in a chip bin, which discharged the chips into a blower. The chips were then blown to a cyclone and discharged to a conveyor, which feeds the PSF. The chips were then dropped into a vertical steam tube before being fed into the refiner. During refining, the primary refiner was controlled to have zero pressure at the inlet and 140 kPa in the casing. From the casing, the primary pulp was blown to a cyclone and discharged and collected in drums, and then treated similarly as in the atmospheric refining runs.

Pulp Tests: TAPPI standard was used for brightness tests. Peroxide residuals were measured using standard iodometric titration.

Running the primary refiner with pressurized casing and atmospheric inlet was compared with conventional atmospheric refining in P-RC APMP pulping of aspen and birch commercial wood chips. The results showed that both refining configurations gave similar bleaching efficiency. For some installations, using pressurized

casing can significantly simplify the process, engineering and operation of P-RC APMP process.

Figure 9 presents the chemical conditions used for P-RC APMP pulping of aspen, and brightness results from atmospheric and casing pressurized runs with the primary refiner. Applying similar AP chemical strategies in both cases, and having similar amounts of total chemical consumption (5.2 to 5.4% total alkali, TA, and 3.7 to 3.9% H₂O₂), both the atmospheric and the casing pressurized gave a similar brightness, achieving 84.2% ISO and 84.7% ISO respectively.

The residual pH (8.8 - 9.0) in both cases were slightly higher than ideal (approximately 7.0-8.5) and the H₂O₂ residual (1.5 to 2.0% on o.d. pulp) was also higher than normal (0.5 to 1.0%), suggesting that in both cases the pulp property could be further developed had the chemical treatments been further optimized.

It is worth pointing out that the bleaching efficiency shown in Table 1 (3.7 to 3.9% H₂O₂ and 5.2-5.4% TA consumption to reach 84.2 to 84.7% ISO brightness) is comparable to or better than bleaching efficiency normally observed in H₂O₂ bleaching of TMP or CTMP pulps from aspen.

Figure 10 presents conditions and results from P-RC APMP pulping of the birch. This particular birch chips was slightly more difficult to bleach than the aspen. Using similar AP chemical strategies, the atmospheric and the pressurizing casing again gave similar bleaching efficiency: 3.1-3.2% TA and 3.4-3.6% H₂O₂ to reach 82.4 to 82.6% ISO brightness. In this case, the residual chemicals (0.1-0.2% TA, 0.5-0.6% H₂O₂ and pH of 8) were within ideal H₂O₂ bleaching conditions.

Example Set C

This example set shows, among other things, that when the chemical recipe and distributions are optimized, the alkali peroxide chemicals at refiner chemical treatment stage can be applied at the

intermediate line in a pressurized refiner system to achieve similar bleaching efficiency as P-RC APMP with conventional atmospheric inlet pressure. Because the residence time is very short in a intermediate line, the same process may also be used in a high pressure refining system, 5 for example a refining system operating at 4 bar or higher.

Wood

All the hardwoods (birch and maple) were received in chip form and mixed separately before being further processed. All the softwoods 10 (spruce, pine and softwood blends) were received in log form, and debarked, chipped and mixed prior to further processing.

Chip Impregnation

The wood chips, unless otherwise specified, were impregnated 15 twice with AP chemicals (consisting of sodium hydroxide (NaOH), hydrogen peroxide (H₂O₂), DTPA, Magnesium Sulfate (MgSO₄) and sodium silicate (Na₂SiO₃), utilizing an Andritz 560GS Impressafiner System. In some cases, the RT-Pressafiner was used at the first stage 20 impregnation (steamed at 1.4 bar for 20 seconds before being pressed).

Refining

An Andritz 36" diameter (91 cm) single disc 36-1CP refiner system 25 was used for all pressurized and atmospheric inlet/casing pressurized runs, and an Andritz 36" diameter (91 cm) double disc 401 system was used for all atmospheric refining runs. Typically, except where stated otherwise, the 401 refiner was used for all secondary and tertiary refining.

Process Description

The P-RC, (Preconditioning, following by Refiner Chemical 30 treatment, where AP chemicals are distributed between chip pretreatment and refining stages), process was used in all trial runs. For the runs where AP chemicals were charged at the intermediate line, the pulp discharged from the blow line was covered under a plastic bag in drums to maintain a temperature of 85-95 °C, depending specific refining

energy used at the refiner, the chemical charges, and the nature of the raw materials.

Pulp Tests

5 Canadian Standard Freeness (CSF) was used for all freeness tests and standard Tappi methods were used for all optical property tests (brightness Tappi T218 OM-83, light scattering, and light absorption coefficient Tappi T425 OM-86 (for handsheet Tappi 205 OM-88)).

10 Figure 15 shows the results obtained by applying AP chemicals at either the refiner eye or the intermediate line during the refiner chemical (RC) treatment stage. Birch and maple woods were used in this example. For each wood species, some chemical pretreatment, (preconditioning), was applied on the chips. For birch the chips were treated with 0.3% DTPA at first stage impregnation, and then 0.2%
15 MgSO₄, 4.4% Silicate, 2.8% TA, and 2.8% H₂O₂ at the second stage impregnation. For maple the chips were treated with 0.5% DTPA at first stage impregnation, and then 0.2% DTPA, 0.1% MgSO₄, 2.0% Silicate, 1.6% TA and 2.6% H₂O₂ at the second stage impregnation.
20 The preconditioned chips then received a similar amount of AP chemicals during refiner chemical (RC) treatment stage, but at different points: one at the refiner eye before refining, and another at the intermediate line immediately after refining.

25 For the birch, both series (A1 and A2) used a total of 5.2% H₂O₂ and 4.6% total alkali (TA), and had a similar amount of H₂O₂ residuals (1.0%-1.1%) and final pH (8.9-9.0). The final pH's were relatively high, indicating that a higher brightness would be achieved if a longer retention time was used. The series from AP addition at the refiner eye (A1) had a similar brightness to samples where AP chemicals were added at the intermediate line, A2, for example, 84.8 versus 84.2% ISO. The slight
30 difference in the brightness was likely, at least in part, due to the slight difference in their freeness, 285 mL for the former case and 315 ml for the latter. In terms of chemistry, both series gave similar light absorption coefficients, 0.27 m²/kg from the former and 0.25 m²/kg from the latter.

In the case of the maple wood, adding AP chemicals at intermediate line, A4, actually gave a higher brightness, 81.9% ISO, than that, 79.2% ISO, from applying the AP chemicals at refiner eye, A3. The difference in this case was a combination of the lower freeness, (295 vs. 5 320 mL), and the lower light absorption coefficient, (0.32 vs. 0.5 m²/kg), of the former.

Softwoods, namely spruce and red pine, were also investigated in to examine effects of different AP chemical applications. Figure 16 summarizes the results, and shows again that similar brightness was achieved by applying AP chemicals at either the refiner eye or the 10 intermediate line. In the case of spruce the chips were first impregnated with 0.3% DTPA, 0.05% MgSO₄, 0.7% Silicate, 0.2% TA and 0.5% H₂O₂, and then 0.1% DTPA, 0.08% MgSO₄, 1.8% Silicate, 1.4% TA and 1.9% H₂O₂ at second stage impregnation.. In the case of red pine 15 the chips were treated with 0.4% TA, 0.5% H₂O₂, 0.2% DTPA, 0.04% MgSO₄ and 0.5% Silicate at first impregnation, and 0.4% TA, 0.6% H₂O₂, 0.14% DTPA, 0.05% MgSO₄, 0.4% Silicate at second 20 stage impregnation. For spruce, using similar amounts of AP chemicals, for example see Figure 16, the blow line series, A6, had a similar or slightly higher brightness of, 78.8% ISO, than the, 78.2% ISO, from the series, A5, where the last stage of AP chemicals were applied at the refiner eye. This slight difference of brightness again was likely a result of combined effects from their slightly different freeness, 47 mL vs. 49 mL, and slightly different light absorption coefficient, 0.56 vs. 0.60 m²/kg.

25 In the case of red pine, the blow line series, A8, had a slightly higher brightness, 71.8 vs. 71.2% ISO, lower light absorption coefficient, 0.84 vs. 1.01 m²/kg, but higher freeness, 99 vs. 82 mL, compared to the refiner eye series, A7. As far as its effect on brightness is concerned, in 30 this case, the difference in the light absorption coefficient was likely the difference in their freeness. The amounts of AP chemical treatment were the same for both series.

A softwood blend from spruce and pine was subjected to high pressure refining at the refiner chemical treatment stage as in Figure 17.

In this case, a RT-Pressafiner was used at the first stage impregnation, and Andritz Model 560GS Impressafiner at the second stage. For this chemical treatment 0.4% TA, 0.6% H₂O₂, 0.18% DTPA, 0.03% MgSO₄ and 0.3% Sodium Silicate at 1st stage chip impregnation; 5 0.4% TA, 0.7% H₂O₂, 0.15% DTPA, 0.05% MgSO₄ and 0.4% Sodium Silicate at 2nd stage chip impregnation; 0.9% TA, 1.5% H₂O₂, 0.18% DTPA, 0.09% MgSO₄ and 1.8% Sodium Silicate at refiner chemical treatment stage, either at the refiner eye as for A9, or the intermediate line as for A10 was used. Series, A9, A10, were 10 performed, and both had similar chemical charges and recipe, but one (A9) had 2.1 bar pressure in the primary refiner and the other, A10, 4.2 bar. Figure 17 presents results, and shows that the series with the higher pressure, A10, was able to achieve similar bleaching efficiency and brightness (using 1.7%TA and 2.8% H₂O₂ and reached 73.7-73.4% ISO). 15 The samples had similar light absorption coefficient (0.96-1.1 m²/kg). These results indicate that when the chemical strategies were optimized, a similar bleaching efficiency and brightness (at least in the range of 70-75% ISO) can be achieved at even a very high pressure (4.2 bar, or 60 psi). The high pressure refining would make it possible to recover high 20 quality steam with better efficiency than the lower pressures, and provide an opportunity to reduce shives (fiber bundles) for high freeness pulps.